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**THERMAL DIFFUSIVITY AND  
HEAT CAPACITY MEASUREMENTS AT  
LOW TEMPERATURES BY THE FLASH METHOD**

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ADMINISTRATIVE INFORMATION

This report covers a facet of the work authorized during FY 1961 and FY 1962 by the Bureau of Ships under RDT & E Subproject S-RO11 01 01, "Fundamental Laboratory Program," Task 0401. Details of this work are found in the U. S. Naval Radiological Defense Laboratory FY 1962 Technical Program as Program A-6, Problem 9, entitled "Thermal Conductivity of Thermoelectric Materials" the objective of which is to investigate the mechanism of heat conduction in thermoelectric materials, and in particular, to determine the separate contributions of the phonons, electrons and photons. Funds to support this work during FY 1962 were provided by the Bureau of Ships on Budget Project 10, Allotment 178/62.

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## ABSTRACT

A method for measuring heat capacity and thermal diffusivity of solid materials from  $-180^{\circ}\text{C}$  to  $+200^{\circ}\text{C}$  is described in this report. A high intensity short duration light pulse is absorbed in the front surface of the specimen and the resulting temperature rise of the rear surface is measured by a thermocouple, displayed on an oscilloscope and photographed by a Polaroid Land camera. From the shape and magnitude of the temperature traces thus obtained, the thermal diffusivity and heat capacity of the specimen are determined. Thermal properties of aluminum alloy No. 2024 and single crystal magnesium oxide were determined with this method over the temperature range from  $-180^{\circ}\text{C}$  to  $+200^{\circ}\text{C}$ .

## SUMMARY

### The Problem:

The need for a reliable method of measuring heat capacity and thermal diffusivity of solid materials at high temperatures has resulted in the development of the flash technique. The objective of this study is to describe an extension of this method to low temperatures and its applicability to transparent solid materials.

### The Findings:

Satisfactory results were obtained with this method for transparent and non-transparent solid materials for temperatures as low as  $-180^{\circ}\text{C}$ .

## INTRODUCTION

Various methods, based on the heat flow equation, are presently used for determining the thermal properties of solids. The heat flow equation can be solved for a wide variety of boundary conditions and the accuracy of the method employed depends on how well these boundary conditions can be met in practice. Two of the boundary conditions not easily satisfied are negligible thermal contact resistance between the specimen and its heat source and negligible surface heat losses during the measurement period. In the flash technique, developed at NRD<sup>1</sup> and employed here, the contact resistance is nonexistent, since the specimen is radiantly heated by a flash tube several centimeters away. Furthermore, surface heat losses are reduced to a minimum by making the measurements in a very short period of time during which the conductive heat losses are negligible.

The flash technique employs a high intensity, short duration flash of radiant energy to abruptly raise the front surface temperature of the specimen under investigation. The resulting temperature history of the rear surface is measured by a thermocouple and the information is amplified and displayed on an oscilloscope. The rate of rise of the rear surface temperature thus observed, determines the thermal diffusivity and the maximum value of temperature attained at the rear surface, gives the heat capacity of the specimen.

It is the purpose of this report to describe an extension of the temperature range of the flash technique to low temperatures and to

show applicability of method in determining thermal properties of transparent solid materials such as single crystal magnesium oxide.

#### THEORY OF THE METHOD

For a perfectly insulated specimen, of uniform thickness, uniformly irradiated across its front surface by a high intensity light pulse of short duration as compared to the time required for the heat pulse to travel through the specimen, the back surface temperature history can be represented by:

$$T(L_1 t)/T_m = 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp(-n^2 \pi^2 \alpha t / L^2) \quad (1)$$

where:

$T(L_1 t)$  = The instantaneous back surface temperature rise at time  $t$ .

$L$  = Thickness of specimen in cm.

$T_m$  = Maximum back surface temperature rise in  $^{\circ}\text{C}$ .

$\alpha$  = Thermal diffusivity in  $\text{cm}^2/\text{sec}$ .

At time  $t_{1/2}$ , the time required for the back surface to reach half of the maximum temperature rise,  $T(L_1 t)/T_m = 1/2$ . Substituting in equation (1) and solving for the thermal diffusivity

$$\alpha = \frac{1.37L^2}{\pi^2 t_{1/2}} \quad \text{cm}^2/\text{sec} \quad (2)$$

The heat capacity,  $C_p$ , is given by:

$$C_p = \frac{Q}{L \Delta T_m} \quad \text{cal/gm}^{\circ}\text{K} \quad (3)$$



$Q$  = Radiant energy from flash lamp in  $\text{cal/cm}^2$

$D$  = Density of specimen in  $\text{g/cc}$

Thermal conductivity is given by equation (4):

$$K = \alpha DC_p \quad \text{cal/cm/sec/}^\circ\text{K} \quad (4)$$

#### EXPERIMENTAL PROCEDURE

Thermal properties of aluminum alloy No. 2024 and single crystal magnesium oxide were evaluated on the experimental apparatus shown in Figure (1). Spring clips secure the specimen in holder (H) which fits in the center of a metal container (C). With the exception of openings (A) and (B) leading through "O" rings to the outside of vacuum chamber (V), container (C) was completely enclosed and could be filled with liquid nitrogen to cool the specimen. A vertical heater coil inside holder (H) and directly beneath the specimen allowed the specimen to be heated. Ten mil chromel-alumel thermocouple wires (TT) passed through the center of the heater coil making contact with the rear surface of the specimen. This thermocouple extended through Kovar seals in the vacuum chamber wall (V) to an ice bath cold junction and then to the amplifier and oscilloscope. Pressure supplied by the spring clips, holding the specimen, maintained the specimen to thermocouple contact. Figure (2) is a picture of the experimental setup.

In order to obtain correct temperature rise data, the area of contact between the sample and the thermocouple wires should be small however, if the wires are too thin, poor contact will result at the high and low temperatures. The sharp ends of the thermocouple wires were separated a few millimeters so that continuity depended on contact through the rear surface of the specimen. To increase their rigidity, the wires were clamped a few millimeters from their ends in a thermocouple clamp,

shown in the detailed drawing of Figure (1), which could be moved vertically through a small opening in the center of the holder top and could be secured with four set screws in the side of the holder. Steady state specimen temperatures were measured with a Rubicon potentiometer. Extreme care was taken to provide good electrical shielding for the entire system in order to minimize noise pickup.

Signal voltages, in the range of .02 to .10 mv, obtained in the output of the chromel alumel thermocouple, were amplified by a differential amplifier having a gain of 380 before being displayed on the oscilloscope.

Originally specimen holder (H) Figure (1), was constructed of lavite in order to reduce conductive heat losses. Due to the poor heat conductive properties of lavite, the low temperature range of the system was somewhat limited and at high temperatures, the high temperature differential existing between the specimen and the nichrome heater coil beneath it, would result in the evaporation and deposit of an electrically conductive film of nichrome on the surrounding surfaces. To remedy this defect, a holder was constructed of boron nitride, a binary nitride of good thermal conductivity, and high dielectric constant. Boron nitride increases the heat losses of the specimen during irradiation but this effect is negligible if the contact area between specimen and holder is kept low.

Uniform irradiation by a short pulse was obtained with a General Electric FT 524 Xenon flash lamp mounted above the vacuum chamber with a quartz window between the specimen and the lamp. The Xenon flash pulse width is 500 $\mu$  sec, considerably smaller than the rise time of the back surface temperature of specimens investigated. A constant voltage maintained across the Xenon flash lamp insured a constant light output. Uniform and constant absorptance to the flash lamp irradiance was

achieved by a thin coat of Parson's black applied on the front surface of all specimens.

Excessive amounts of Parson's black affected the shape of the rear surface temperature rise, especially with specimens of high diffusivity, but the effect was negligible when the amount of black was a small fraction of a percent of the specimen's weight.

Single crystal magnesium oxide is a transparent solid with very poor electrical conductivity. In order to obtain a good conductive surface where the thermocouple made contact with the specimen, Hanovia liquid platinum coatings were applied on the specimen's rear surface. Parson's black was applied on the specimens front surface for constant absorptivity. In equation (1), it is assumed that all radiant energy absorbed by the specimen is absorbed at its front surface. Parson's black, however, does not provide a totally opaque surface and although with non-transparent specimens, this results in the reflection of a small and practically constant fraction of the incident radiant energy, in magnesium oxide, the radiant energy normally reflected passed through the specimen and raised the temperature of the platinum coated rear surface. To alleviate this effect, Hanovia liquid platinum was applied on the specimen's front surface for opaqueness and then painted with Parson's black for constant absorptivity. The effect of the platinum coating is assumed to be very small since the heat capacity of platinum is low as compared to that of magnesium oxide and the thickness of the coating is negligible in comparison to the magnesium oxide specimen thickness.

Figure (3) is typical of the rear surface temperature rise as observed on an oscilloscope. Knowing the gain of the system, the sweep time of the oscilloscope and the time the flash lamp is triggered, indicated by a transient on the base line of the trace in Figure (3), the maximum temperature rise  $T_m$ , is represented by the maximum deflection

of the trace from the base line and time  $t_{1/2}$  by the time required for the trace to rise to  $1/2 \times T_m$  from the instant the flash lamp is triggered.

The  $T_m$  and  $t_{1/2}$  parameters were used in equations (2) and (3) to determine the thermal diffusivity and heat capacity of each specimen over a range of temperatures.

## RESULTS

The boundary conditions on which equation (1) is based require a thermally well-insulated specimen to be uniformly irradiated by a high energy short duration pulse. In Figure (4), the solid line is a theoretical curve of the rear surface temperature history obtained from equation (1) and any data deviating from this curve indicates a departure from the above boundary conditions. The points in Figure (4) are from an actual temperature rise curve for magnesium oxide. At the knee of the curve, the discrepancy is attributed to the effects of the platinum and Parson's black coatings but this does not affect the results obtained since the two plots agree at the  $t_{1/2}$  and  $T_m$  points and the maximum deviation between the two plots is less than 5%.

Two specimens, one copper .078" and one silver .039" thick, of known thermal capacities over the temperature range from  $-180^{\circ}\text{C}$  to  $+200^{\circ}\text{C}$  were irradiated for the purpose of determining the value of  $Q$  in equation (3). Figure (5) is a plot of  $Q$  versus temperature for the above two specimens. From Figure (5) it is seen that  $Q$  remains constant at  $0.129 \text{ cal/cm}^2$ , indicating the absorptivity of Parson's black, applied on all front surfaces, remains constant over the temperature range. The value of  $Q$  is dependent on flash lamp voltage, lamp to specimen distance and front surface absorptivity of specimen. If the

above conditions remain unchanged,  $Q$  is a constant. However, its value was periodically checked using copper and silver specimens, at room temperature only, and all values obtained agreed within 5% of the original value obtained from Figure (5).

Figure (6) is the heat capacity curve of aluminum alloy No. 2024 versus temperature from data taken on two specimens, one .031" thick and one .024" thick.

Figures (7), (8), and (9) are heat capacity, thermal diffusivity and thermal conductivity curves of single crystal magnesium oxide versus temperature from data taken on two specimens, one .106" thick and one .086" thick.

#### CONCLUSIONS

The close agreement between the theoretical curve and experimental points in Figure (4) indicate that the theoretical boundary conditions of equation (1), i.e., low thermal losses, constant absorptivity and uniform irradiation by a short duration light pulse, were met satisfactorily.

Accuracy of data obtained with the flash technique is well within 5%. The high voltage applied to the Xenon flash lamp was kept constant within 1%, amplifier gain was measured within 1%, traces observed were measured from the Polaroid prints within a 2% accuracy and the thermocouple used was checked at 0°C, -78°C, and -196°C and found to have a maximum error of less than 2% at -196°C.

The largest data fluctuation observed was for magnesium oxide Figure (7), where at the high temperatures, values for heat capacity varied within a 10% range. This is attributed to the platinum

coating on the magnesium oxide, which distorts the uniformity of the front surface and which effect is apparently more pronounced at the higher temperatures. However, the high temperature portion of the heat capacity curve obtained for magnesium oxide agrees within 4% with already published data as indicated in Figure (7). Aluminum alloy No. 2024 consists of 90% aluminum and if the heat capacity curve in Figure (6) were compared to that of pure aluminum, they would be found to agree very closely especially at the low temperature range. All other data repeated itself within 5%.

The good degree of repeatability obtained with the known and unknown specimens in the report, demonstrates the correctness of the method employed, degree of accuracy expected and that Parson's black paint will provide a front surface of constant absorptivity to irradiation with the spectral distribution of the Xenon lamp, independent of temperature but within the stability range of the paint. This paint has shown satisfactory stability in the range from  $-196$  to  $+160^{\circ}\text{C}$ .

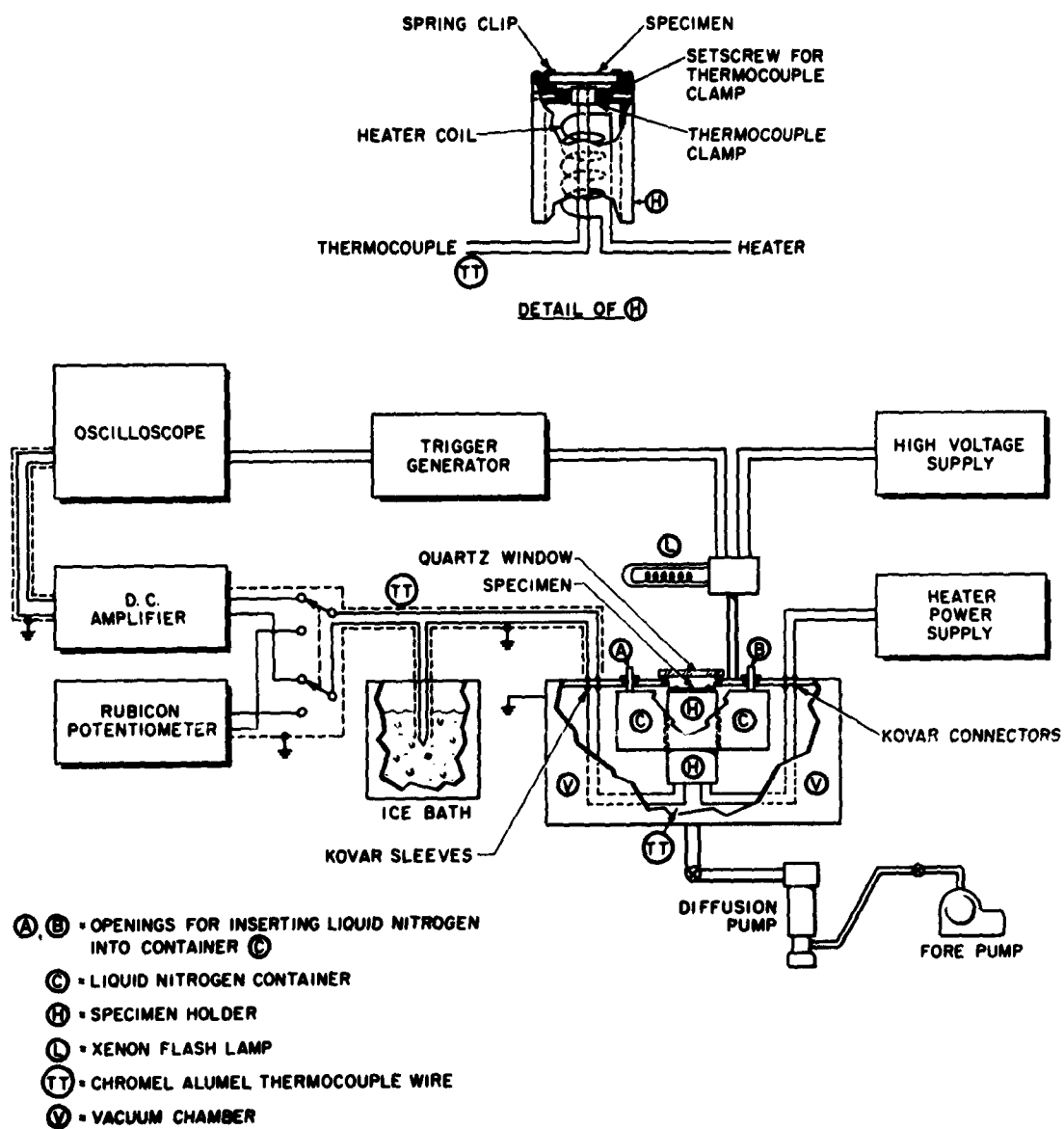


Figure 1 Block Diagram of Experimental Arrangement

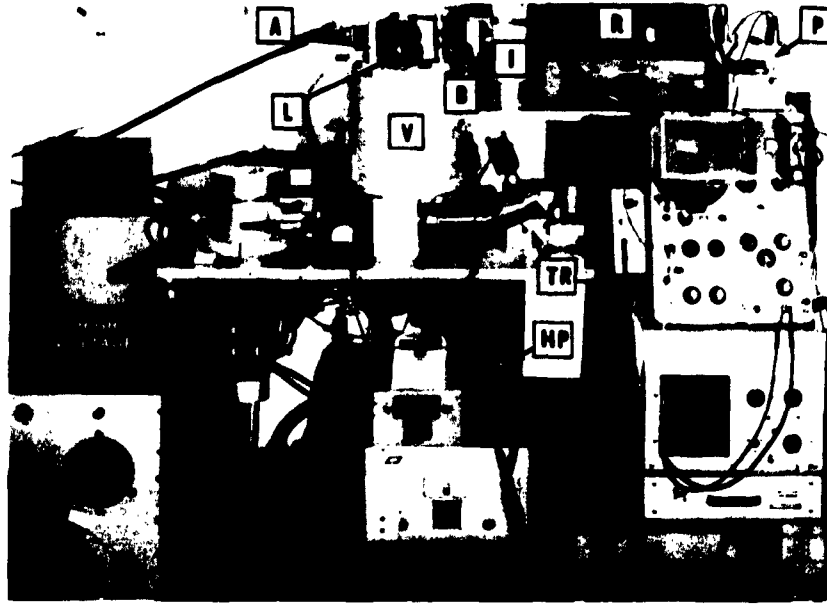


Figure 2 Photograph of Experimental Setup. (A),(B) = Openings for inserting liquid nitrogen, (HP) = Heater power supply variac, (I) = Ice bath, (L) = Xenon flash lamp, (P) = D. C. Amplifier, (R) = Rubicon potentiometer, (TR) = Trigger generator, (V) = Vacuum chamber

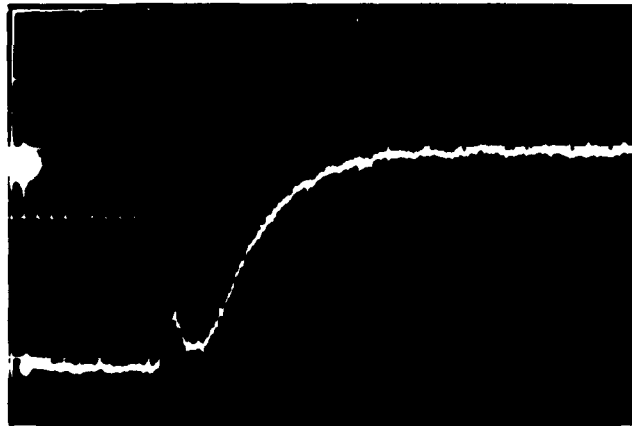


Figure 3 Photographic Trace of Rear Surface Temperature Rise



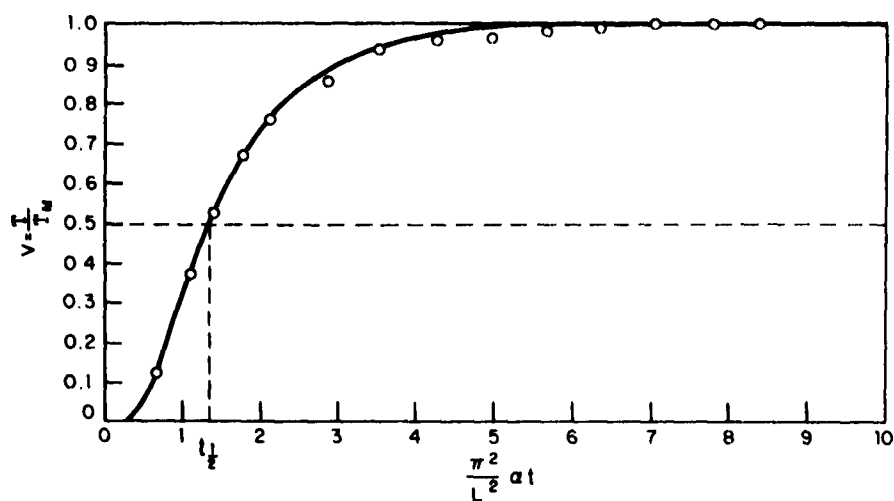


Figure 4 Points of Observed Temperature Rise Curve Plotted Against Theoretical Rear Surface Temperature Curve from Equation (1)

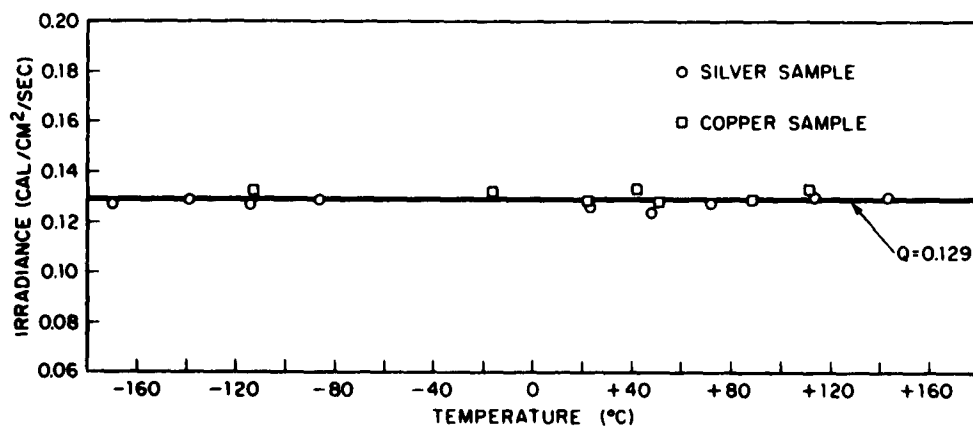


Figure 5 Irradiance of Xenon Flash Lamp

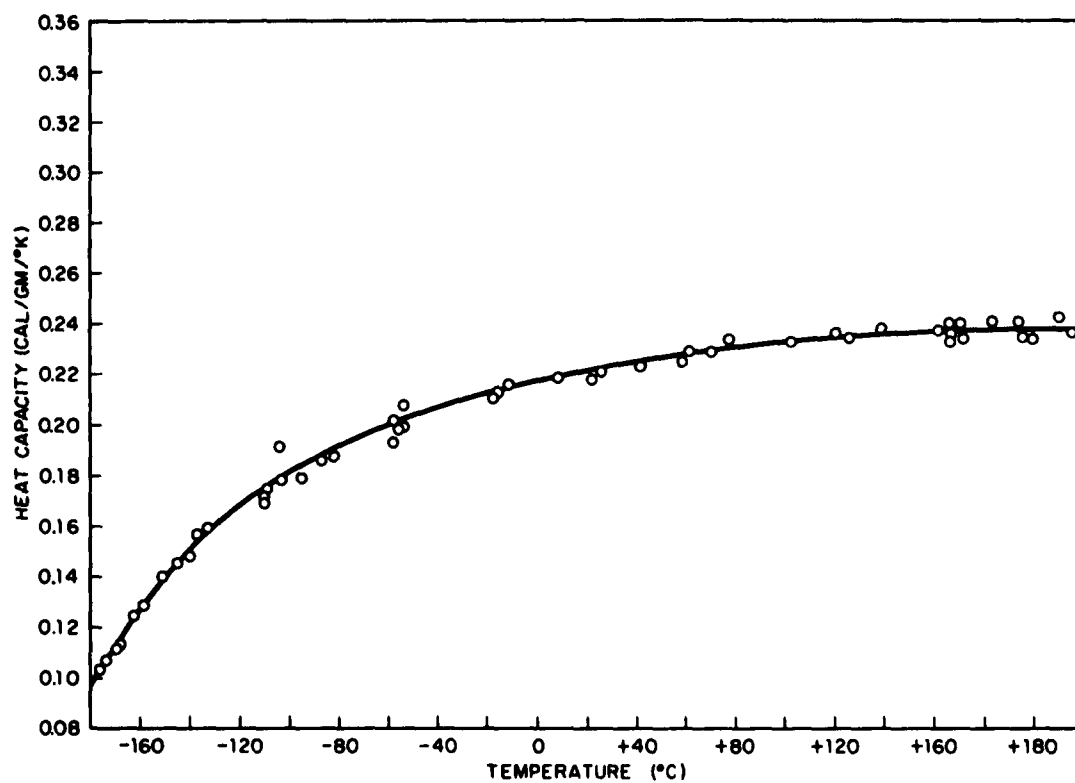


Figure 6 Heat Capacity of Aluminum Alloy No. 2024

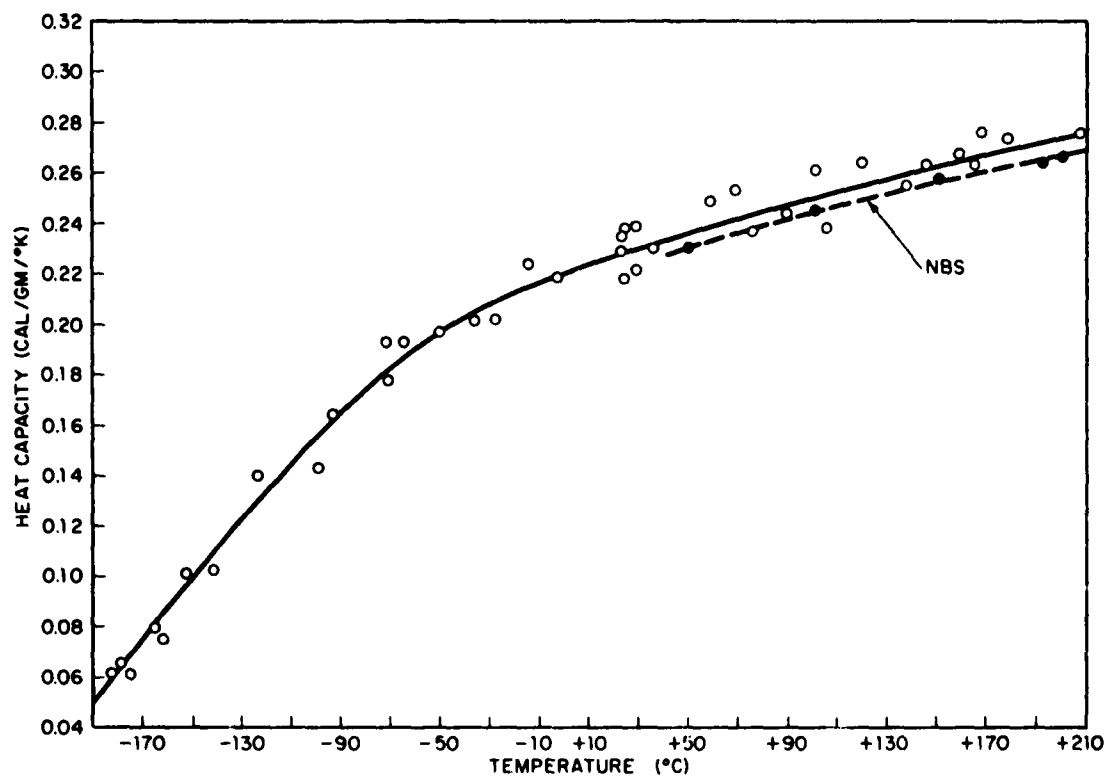


Figure 7 Heat Capacity of Single Crystal Magnesium Oxide

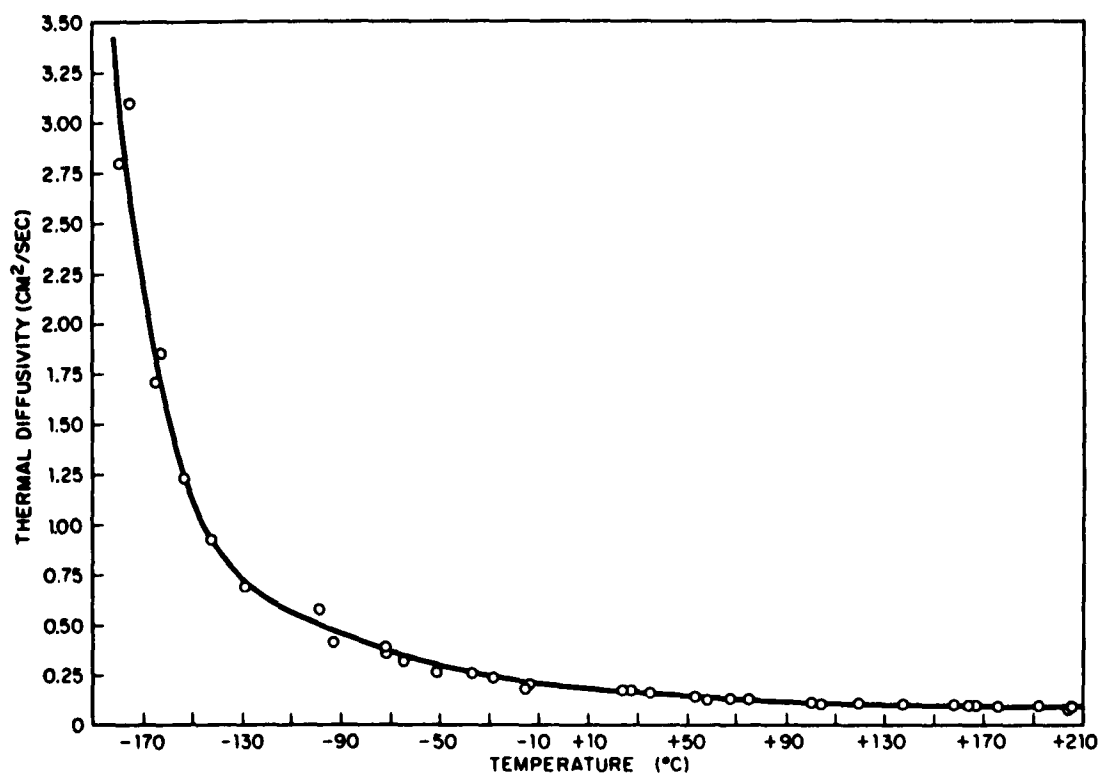


Figure 8 Thermal Diffusivity of Single Crystal Magnesium Oxide

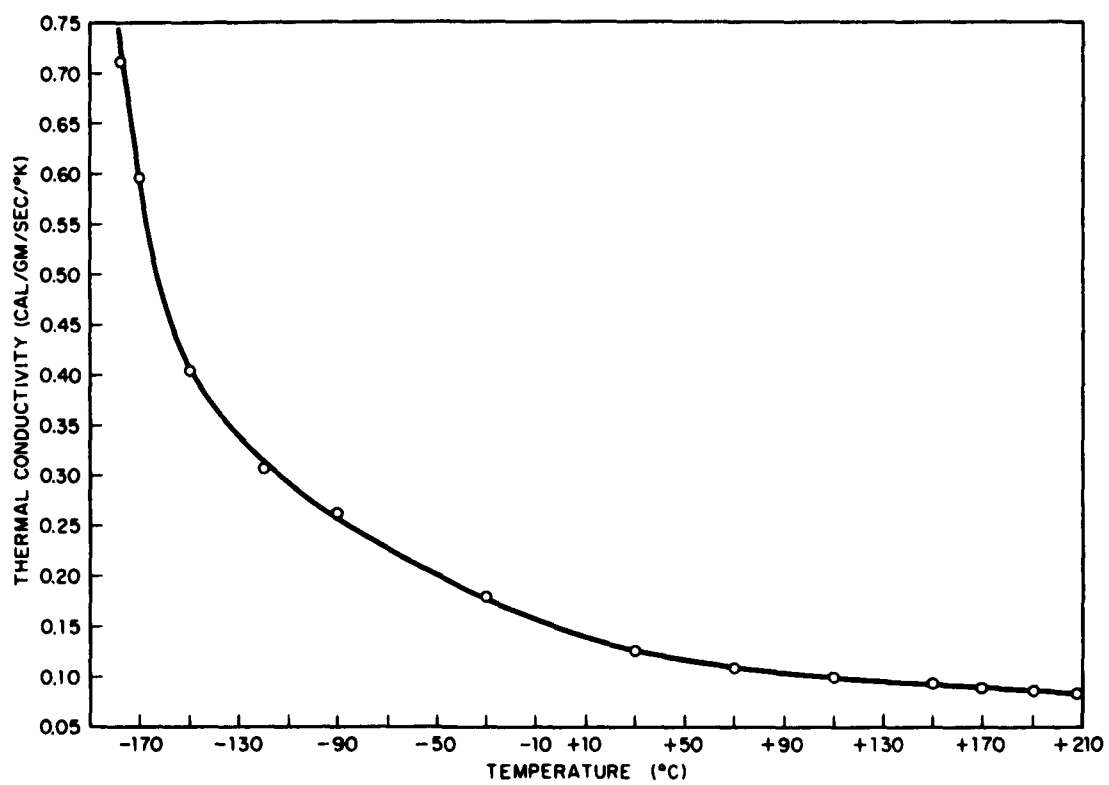


Figure 9 Thermal Conductivity of Single Crystal Magnesium Oxide

#### REFERENCES

1. W. J. Parker, R. J. Jenkins, C. P. Butler, G. L. Abbott, Flash Method of Determining Thermal Diffusivity, Heat Capacity, and Thermal Conductivity J. Appl. Phys., Vol. 32, No. 9, 1679

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